

Status Report

DEVELOPMENT OF NOVEL EOR METHODS
Microbial Technology

Project BE14, Milestone 5, FY88

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ABSTRACT

NIPER Project BE14, Development of Novel EOR Methods, encompasses a series of experiments designed to develop cost-effective methods for improving oil recovery. Microbial EOR is the least expensive of the chemical flooding processes, and with today's economic climate, offers a potentially cost-effective EOR process. This status report describes experiments performed using microorganisms and several different chemical and nutritional additives for improving the oil recovery efficiency of the microbial formulation. In this first year of the work, a number of different EOR chemicals were combined with microbial formulations in corefloods and flask tests. The project research will be continued in FY89 with NIPER Project BE3, Improved Microbial Flooding Methods.

INTRODUCTION

The biological research for Project BE14, Development of Novel EOR Methods, has emphasized studies to determine if combinations of chemicals and microbial solutions could be used for improved oil recovery. Laboratory experiments were designed and conducted to establish benefits and constraints of adding chemicals (low concentrations of surfactants, dilute solutions of polyacrylamides, and/or lignosulfonates) to the lowest cost chemical EOR process, microbial flooding. The hypothesis was that enhancement of the microbial flood residual oil recovery could occur by adding a small pore volume of chemical additive.

EXPERIMENTAL

A series of compatibility tests was conducted with the microbial formulation NIPER Bac 1 (see table 1), and the petroleum sulfonate Witco TRS 10-410. The microorganisms were inoculated in trypticase soy broth (nutrient) containing varying concentrations of TRS 10-410 (table 2). The results shown in table 2 indicate that the microorganisms grew better in the 1:1 rather than in the 1:10 mixture of microorganisms to surfactant. Only NIPER 2, a *Bacillus*

species, showed inhibited growth at the higher surfactant concentration. Microbial compatibility tests were conducted concurrently using brine and Delaware-Childers crude oil used for microbial coreflooding experiments.

It was concluded that TRS 10-410, at a concentration of 0.2% or less is not bactericidal; when present at a ratio of 10:1 with the bacteria, it is somewhat bacteriostatic. At concentrations of 0.05 to 0.2%, TRS 10-410 may even stimulate growth of the bacteria.

Studies were conducted to determine the importance of proper handling, screening, and injection procedures in the design of microbial enhanced oil recovery (MEOR) projects. This was accomplished by waterflooding 1- and 4-ft cores while altering the slug size, molasses concentration, incubation period, flood rate, sequence of injection, and time and volume of additional feedings. The tests were made using oil-saturated cores that were brine flooded to residual oil saturation. It was observed that the incubation period between the time of injection and the time of the waterflood is one of the most important parameters. When the microorganisms are shut in after injection for 3 days, oil recovery increases (see figure 1). Core B28 was injected continually with molasses, and the oil recovery efficiency of the microbial culture increased at the same rate and leveled out (fig. 2). When the concentration of molasses was increased from 4 to 10%, other microorganisms were stimulated that produced polymer and caused a reduction in brine permeability without recovering additional oil. Such findings emphasize the importance of process design in MEOR applications.

Three microbial corefloods were conducted to evaluate the effects of adding a low concentration of TRS 10-410 with the microbial formulation. The results are presented in table 3. The addition of the surfactant appeared to have no effect on the residual oil recovery efficiency of the microbial formulation. When the experiment was repeated with a concentration of 0.5% sodium lignosulfonate, no oil recovery was observed. Later compatibility testing showed that this concentration, as well as a lower concentration of 0.1%, was inhibitory to NIPER Bac 1.

Three additional microbial corefloods were conducted in order to determine if a combination of microorganisms that produced surfactant could enhance oil recovery by the use of a polymer solution behind the microbial surfactant (table 4). Three cores, MP1, MP2, and MP3, were flooded to residual oil

saturation with Delaware-Childers crude oil and 0.5% sodium chloride brine. The cores had pore volumes of 145 ml and permeabilities of about 400 md. Cores MP1 and MP2 were injected with 0.1 PV of a NIPER 1 and NIPER 3 microbial mixture and 0.2 PV of 4% molasses. Both cores were shut-in for 3 days and then re-fed with 0.2PV of molasses, shut-in for another 3 days, re-fed one more time, and then waterflooded. Core MP1 was flooded with 0.1% K5D polymer (viscosity of 35.6 cP, Kelco), while core MP2 was flooded with 0.5% NaCl brine. The residual oil recovery efficiency for MP1 was 16.3% and for MP2, 13.2%. Core MP3 was a control core which was not microbially treated and only flooded with 0.1% K5D polymer (viscosity of 35.6 cP). MP3 recovery efficiency was 3.6%. This set of corefloods showed that addition of polymer to the waterflood increased oil recovery efficiency by the same amount that a polymer flood alone would yield, approximately 3%. No synergism was observed using the polymer in the waterflood. Repetition of this experiment in parallel cores of high and low permeabilities would be very enlightening.

Concurrent studies were carried out using several different microbial species and the biopolymer, K5D. This polymer was chosen because it contains no biocides (such as formaldehyde) which might inhibit microbial growth. The purpose of these experiments was to determine if a microbial solution could break down a polymer and produce a surface-active agent at the same time. At first, only growth experiments were performed to find microbes that would grow in the presence of the polymer (table 5). A set of growth experiments was performed, and the surface tension was measured (table 6). Finally, the solution viscosity and surface tension were both measured in microbial/biopolymer growth experiments (table 7). Results from these studies showed that the microbial surfactant was unaffected by the addition of polymer, and the viscosity of the polymer did not decrease.

RESULTS AND CONCLUSIONS

The combination of the petroleum sulfonate, TRS 10-410, and the microbial formulation did not appear to synergistically improve oil recovery. Other surfactants may yield different results. The use of a microbial formulation, followed by a polymer flood, did not synergistically improve oil recovery from 10-in. cores. It would be useful to test this process in some type of fractured core or parallel cores with highly different permeabilities to

determine if an improvement in sweep efficiency by use of the polymer would improve oil recovery with the microbial system. A few microbial isolates were identified that could metabolize and grow using the biopolymer K5D. The growth in K5D was not as extensive when compared with growth in a more traditional microbial medium, such as trypticase soy broth. We observed that one of the microorganisms was able to lower the surface tension of the K5D solution, and it may be possible to enhance this ability for investigations relating to polymer combinations with microbial formulations.

TABLE 1. - Descriptions of microorganisms used in BE14 studies

BAC 1 - A mixture of NIPER 1, 2, 3, and 4

Bacillus licheniformis - NIPER 1

Bacillus sp. - NIPER 2

Clostridium sp. - NIPER 3

Gram-negative facultatively anaerobic rod - NIPER 4

Clostridium sp. - NIPER 6

TABLE 2. - Compatibility tests with NIPER Bac 1 and TRS 10 surfactant

1:10 Tubes ¹	- 0.2% TRS 10-410 - 2+ growth, lots of spores, NIPER 2 seen
	0.1% TRS 10-410 - 3+ growth; lots of spores, all bacteria seen
	0.05% TRS 10-410 - 2+ growth; lots of spores, all bacteria seen
	0.01% TRS 10-410 - 2+ growth; NIPER 2 seen
1:1 Tubes ²	- 0.2% TRS 10-410 - 4+ growth; all bacteria seen, lots of spores
	0.1% TRS 10-410 - 4+ growth; all bacteria seen, less spores than above
	0.05% TRS 10-410 - 4+ growth; all bacteria seen, few free spores
	0.01% TRS 10-410 - 3+ growth; mostly <i>Bacillus</i> spp. and M18 (NIPER 1 & 2 and NIPER 4, only a few spores present)

¹Test tubes contained a 1:10 mixture of bacteria (grown in trypticase soy broth (TSB):TRS 10-410 (conc. of 0.2%, 0.1%, 0.05%, and 0.01%).

²Test tubes contained a 1:1 mixture of bacteria and TRS 10-410 (same conc. as above).

Tubes were incubated anaerobically at 30° C for 6 days.

4+ = excellent growth.

2+ = little growth.

TABLE 3. - Microbial/surfactant coreflood results

Core	Injectant	k	S _{owf}	S _{ocf}	Er	Pressure psi	Microbial CFU/ml	
							Aerobic	Anaerobic
MS1	0.03 PV BAC 1	278	32.5	29.7	8.6	30	7.9 X 10 ⁵	5.7 X 10 ⁴
	0.03 TRS 10-410							
MS2	0.05 PV BAC 1	256	34.9	32.6	9.3	25	2.3 X 10 ⁵	1.7 X 10 ⁵
	0.05 TRS 10-410							
MS3	0.03 PV BAC 1	425	35.4	32.1	9.3	40	1.6 X 10 ³	1.3 X 10 ³

Unfired Berea, Hassler coreholders, cores were injected with an equal PV of molasses, shut in 3 days, then re-fed with the same amount of molasses, shut in 3 days, then waterflooded.

Nutrient was OKC molasses, 4% concentration.

Bac 1 = Mixed culture of NIPER 1,2,3, and 4.

k = absolute permeability to brine in millidarcies.

S_{owf} = residual oil saturation after waterflooding (% PV).

S_{ocf} = residual oil saturation after microbial treatment (% PV).

Er = recovery efficiency $\frac{S_{owf} - S_{ocf}}{S_{owf}} \times 100\%$.

psi = maximum increase in pressure during core incubation.

CFU/ml = Colony forming units/ml.

TABLE 4. - Microbial/polymer core experiments

Core	Injectant	k	S _{owf}	S _{ocf}	Er	psi
MP1 ¹	NIPER 1 & 6	434	35.5	29.7	16.3	45
MP2	NIPER 1 & 6	394	37.8	32.8	13.2	45
MP3 ¹	Polymer only	421	39.2	37.8	3.6	-

¹MP1 and MP3 were flooded with 0.1% K5D Polymer at 35.6 centipoise viscosity; while MP2 was flooded with 0.5% NaCl brine, all at 1 ft/d.

Nutrient was OKC molasses, 4% concentration.

k = absolute permeability to brine in millidarcies.

S_{owf} = residual oil saturation after waterflooding (% PV).

Er = recovery efficiency $\frac{S_{owf} - S_{ocf}}{S_{owf}} \times 100\%$.

S_{ocf} = residual oil saturation after microbial treatment (% PV).

psi = maximum increase in pressure during core incubation.

TABLE 5. - Microbial growth ratings with polymer

Microbe	0.1% K-5D(W)	0.5% K-5D(W)	1%K-5D(W)	0.1%K-5D(B)	0.5%K-5D(B)	1%K-5D(B)
NIPER 5	1-	1-	1-	4+	4+	4+
NIPER 3	1-	0	0	4+	4+	4+
NIPER 2	1-	1+	1-	1+	1+	2+
NIPER 1	1-	1-	1-	2+	3+	3+
POLYMER 1	1-	1-	1-	2+	2+	3+
POLYMER 4	1-	1+	1-	2+	2+	2+
BAC 1	1-	1-	1-	4+	4+	4+
CONTROL	0	0	0	0	0	0
POLYMER - K-5D (XANTHAN GUM) - LOT #280044						

(W) = Water only (B) = trypticase soy broth.

Solutions of 0.1%, 0.5%, and 1.0% conc. of K-5D were prepared in sterile water (W) and sterile trypticase soy broth test tubes (B) (10 mL each).

Bacterial cultures were inoculated and incubated in the anaerobic glovebox at 30° C for 1 wk.

4+ = excellent growth.

0 = No growth.

TABLE 6. - Surface tensions of spent media from previous experiments

		Surface Tension dynes/cm
Controls	TSB - 0.1%K-5D	57.3
	TSB - 0.5%K-5D	57.0
	TSB - 1% K-5D	56.5
	0.5% K-5D	66.5
	1.0% K-5D	67.0
NIPER 3	TSB+0.1%K-5D	54.0
	" 0.5%K-5D	52.0
	" 1.0%K-5D	55.0
NIPER 5	TSB+0.1%K-5D	46.0
	" 0.5%K-5D	50.0
	" 1.0%K-5D	46.0
NIPER 2	TSB+0.1%K-5D	49.0
	" 0.5%K-5D	46.0
	" 1.0%K-5D	44.0
NIPER 1	TSB+0.1%K-5D	48.0
	" 0.5%K-5D	32.0
	" 1.0%K-5D	48.0
POLYMER 1	TSB+0.1%K-5D	49.5
	" 0.5%K-5D	50.0
	" 1.0%K-5D	48.0
POLYMER 4	TSB+0.1%K-5D	48.0
	" 0.5%K-5D	49.0
	" 1.0%K-5D	51.0
BAC 1	TSB+0.1%K-5D	56.0
	" 0.5%K-5D	55.0
	" 1.0%K-5D	54.0
NIPER 2 + 0.5% K-5D(W)		64.0
POLYMER 4 + 0.5%K-5D(W)		57.0
NIPER 1 + 0.5%K-5D(W)		64.0
NIPER 1 + 1%K-5D(W)		64.0

TSB = Trypticase soy broth

TABLE 7. - Results of repeat of previous experiments (table 6) with viscosity and surface tension measurements

Controls	Growth	Surface tension	Viscosity, cP
0.01% K5D	-	68.0	3.58
0.05% K5D	-	67.0	8.37
0.05% K5D (TSB)	1+	54.0	8.60
0.01% K5D (TSB)	1+	52.0	3.98
0.01% NIPER 2	1-	62.0	-
0.05% NIPER 2	1-	64.0	-
0.01% (TSB) NIPER 2	3+	50.0	3.86
0.05% (TSB) NIPER 2	3+	53.5	-
0.01% NIPER 1	0	66.0	-
0.05% NIPER 1	1+	43.0	7.85
0.01% (TSB) NIPER 1	3+	28.0	3.96
0.05% (TSB) NIPER 1	4+	26.0	8.77
0.01% NIPER 5	1-	63.0	-
0.05% NIPER 5	1+	59.0	7.89
0.01% NIPER 5 (TSB)	4+	45.5	3.77
0.05% NIPER 5 (TSB)	4+	52.0	-
0.01% POLY 4	1-	65.0	-
0.05% POLY 4	1+	62.0	-
0.01% POLY 4 (TSB)	4+	48.0	3.94
0.05% POLY 4 (TSB)	4+	47.0	8.79

4+ = excellent growth.

0 = no growth.

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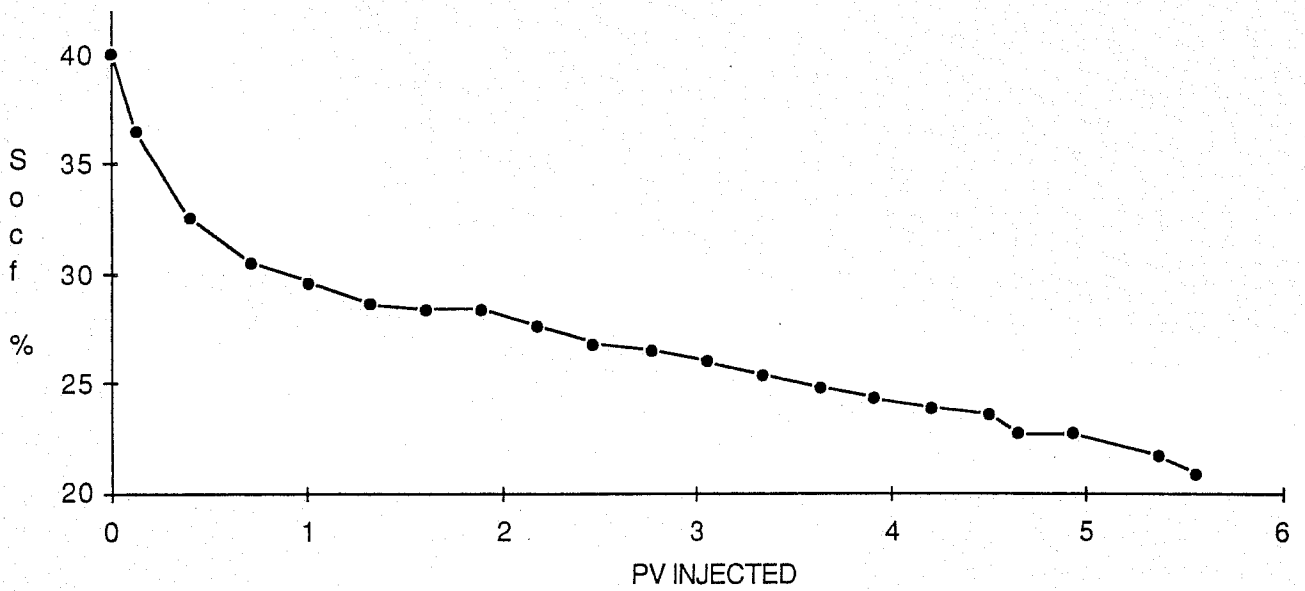


FIGURE 1. - Reduction of residual oil saturation (S_{ocf}) by microbial treatment.

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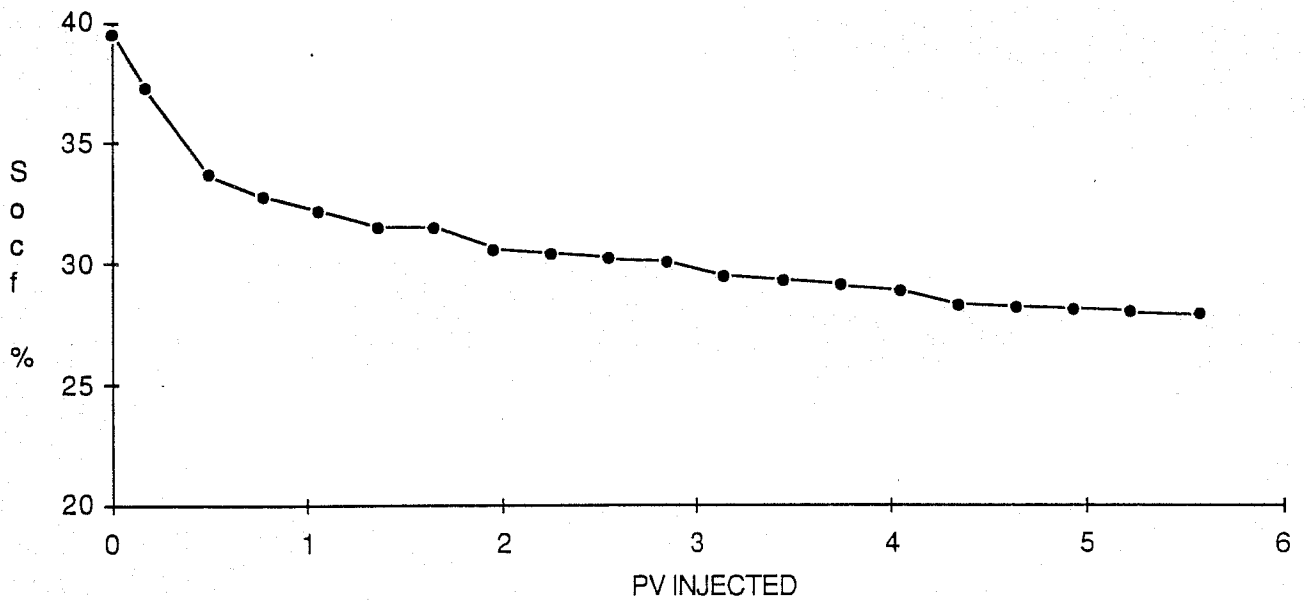


FIGURE 2. - Reduction of residual oil saturation (S_{ocf}) using continuous molasses in the waterflood.